TECHNICALLY speaking

BY PATRICK VALENTINE, NORTH AMERICAN PCB AND EP&F BUSINESS MANAGER, OMG ELECTRONIC CHEMICALS, MAPLE PLAIN, MINN.

Using the Plackett-Burman Family of Designed Experiments for Troubleshooting Process Controls

In 1946, R.L. Plackett and J.P. Burman published their now famous paper "The Design of Optimal Multifactorial Experiments" in *Biometrika* (vol. 33). The paper described the construction of economical designs with the run number a multiple of 4, rather than a power of 2, like full factorial and fractional factorial designed experiments.

Plackett-Burman designs are very efficient screening designs when only main effects are of interest.[1] These designs are used for screening experiments because main effects are, in general, confounded with two-factor interactions.

Designed experiments that are confounded are described by their resolution. A designed experiment that has a main factor confounded with a two-factor interaction is a Resolution III. An easy way to remember this is to hold three of your fingers up in front of you, grab two of your fingers with your opposite hand and you have one finger that is confounded with the two held in your other hand. This simple example can be used with Resolution IV and V designs to visualize the confounding, using four and five fingers respectively.

Another way to look at this is by correlation analysis. With a Resolution III fractional factorial design, the correlation between a main effect and a two factor interaction is either -1 or +1 (remember that correlations range between -1 and +1, and in correlation analysis we usually examine correlations that are ≥ 0.70). An example of this is shown with a 2^3 half fractional design in Figure 1. The (-) and (+) signs represent the low and high factor levels and are coded as such. Summing this up, what we have with fractional factorials (2^{k-p}) Resolution III designs, is a main effect column X_i is either orthogonal to X_iX_j (sums to 0) or is identical to minus one (-1) or plus one (+1) X_iX_j.

Factors			Interactions		
A	B	C	AB	AC	BC
-	-	+	+	-	-
-	+	-	-	+	-
+	-	-	-	-	+
+	+	+	+	+	+

Figure 1. Confounding effects of a Resolution III design with correlation of -1 or +1.

Even though the Plackett-Burman designs are Resolution III, they possess one unique attribute. The two-factor interaction column X_iX_j is correlated with every X_k (for k not equal to i or j) at a correlation value of -0.33 or +0.33; with this weak correlation the effects of confounding are minimized. Furthermore, the interactions are uniformly dispersed over all the experimental runs. These two attributes make the Plackett-Burman designs good choices for screening experiments when the number of factors being studied is ≥5.

Plackett-Burman design runs are a multiple of 4, beginning with 12 runs (there is no 16 run experiment). Each design can have up to k = (n-1) factors; for example, the simplest Plackett-Burman, the 12 run, can have up to 11 factors (11 = [12 – 1]). One can easily see the advantages to a 12 run experiment being able to model up to 2^{11} = 2,048 different

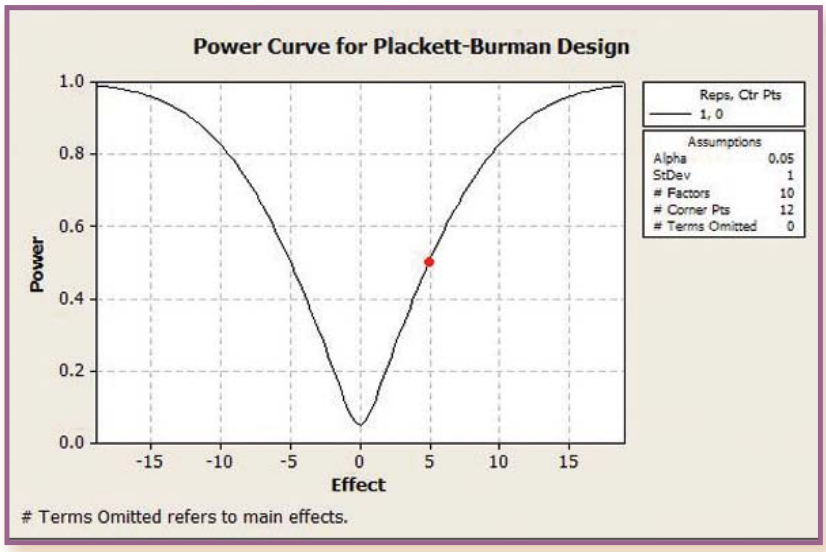

Figure 2. Power curve for the Plackett-Burman 12 run DoE with 10 factors and an alpha level of 0.05.

Factors	-1	+1	Description
Step Exposure	10	15	Stouffer step wedge exposure level for the soldermask
Developer Passes	1	2	Number of passes through the soldermask developer
Pre Cure	No	Yes	Soldermask pre cure time @180°F
Final Cure (min)	15	65	Soldermask cure time @ 300°F
UV Bump	No	Yes	Ultra violet bump of the soldermask prior to ENIG processing
Pre Scrub	Yes	No	Light brush scrub prior to processing the ENIG line
Cleaner	Yes	No	Use of acid cleaner in the ENIG line
Micro Etch (µ-in)	10	90	Micro-inch removal in the etch step in the ENIG line
Pre Dip (min)	1	5	Sulfuric pre dip time prior to the palladium activator in the ENIG line
Activator (min)	1	4	Palladium activator time in the ENIG line

Table 1. DoE factors and levels.

process combinations!

These designs are also known as saturated main effect designs, because all the degrees of freedom are utilized to estimate main effects. If no p-values (probability) are calculated, due to design saturation, then the analysis can be done graphically or by calculating percentages based upon the main effects Sum of Squares divided by the total Sum of Squares. If we apply the principles of Pareto, then we'll most likely be removing multiple main effects, which will, in turn, give us the needed degrees of freedom to calculate the p-values.

If we use a maximum of k = (n-2) factors, this allows one degree of freedom for the error term, which allows the calculation of p-values for each of the main effects in the model simultaneously. Even with this we can still have up to 2^{10} = 1,024 different process combinations.

Saturated Plackett-Burman designs typically have low power. Power is defined as the conditional probability that you will *avoid* a Type II error, and a Type II error is failing to reject (accepting) the null hypothesis when in fact it's false. Ideally we want power to be at least 50% with a preferred power value a ≥80%.

Certainly, as we refine our model by removing non-significant main effects, we'll increase power. Another way to increase power is to increase our risk of making a Type I error, that of rejecting the null hypothesis when, in fact, it is true. And we do that by increasing the alpha (α) value in the significance testing. Figure 2 shows a power curve for a Plackett-Burman 12 run experiment with 10 factors and an alpha value of 0.05. We can see on the horizontal axis that with 50% power we'll need to see an effect size of ±5 standard deviations! And the effect size is the difference between the low and high factor level means. (Discussions on power calculations can get lengthy, so we'll save that for another day.)

Historically, alpha values of 0.05 or 0.10 were used due to computations being done by hand. With the advent of the modern computer we can easily alter the alpha value and determine its effects. For screening experiments I recommend using alpha values up to ~0.15 be used to increase power in the experiment. After all, it's a screening experiment and we don't want to make the acceptance criteria too harsh.

There are six basic steps to follow when designing and running experiments:

1) State the problem, define the objectives
2) Design the Experiment
   a. Choose factors & reasonable ranges for each
   b. Determine appropriate responses & how to measure
   c. Select a design, know your pros & cons, and review runs
   d. Approximate power of the experiment
3) Randomize & run the experiment
4) Analyze the experiment
5) Confirmation runs & P_{pk} estimations
6) Report & recommendations

Figure 3. Stray plating defect seen on blue soldermask.

It does make sense to use the Plackett-Burman family of designed experiments to separate the "vital few" factors from the "trivial many" when troubleshooting a process.

Let's look at a real example using the Plackett-Burman 12 run screening experiment:

Problem statement: During electroless nickel/immersion gold (ENIG) plating of printed circuit boards a random defect is seen that is known as stray plating (plating of nickel and subsequent gold on top of the soldermask). See Figure 3 for an example of stray plating. This defect leads to rework and potential shorts during or after assembly.

After a brainstorming session, the factors and levels were chosen and are shown in Table 1. Concurrently the defect sample in Figure 3 was removed with Scotch® tape and sent to an outside lab for analysis using SARIS™ Material Analysis, which is a combination of high-power laser beam technology used to ablate the sample and subsequent analysis with the high mass resolution capability of the ICP-MS; elemental survey and quantitative analysis of the material

Standard Order	Step Exposure	Developer Passes	Pre Cure	Final Cure	UV Bump	Pre Scrub	Cleaner	Micro Etch	Pre Dip	Activator
1	15	1x	Yes	15	No	Yes	No	90	5	1
2	15	2x	No	65	No	Yes	Yes	90	5	4
3	10	2x	Yes	15	Yes	Yes	Yes	10	5	4
4	15	1x	Yes	65	No	No	Yes	10	1	4
5	15	2x	No	65	Yes	Yes	No	10	1	1
6	15	2x	Yes	15	Yes	No	Yes	90	1	1
7	10	2x	Yes	65	No	No	No	10	5	1
8	10	1x	Yes	65	Yes	Yes	No	90	1	4
9	10	1x	No	65	Yes	No	Yes	90	5	1
10	15	1x	No	15	Yes	No	No	10	5	4
11	10	2x	No	15	No	No	No	90	1	4
12	10	1x	No	15	No	Yes	Yes	10	1	1

Table 2. DoE design array.

Standard Order	Step Exposure	Developer Passes	Pre Cure	Final Cure	UV Bump	Pre Scrub	Cleaner	Micro Etch	Pre Dip	Activator	Stray Plate
1	15	1x	Yes	15	No	Yes	No	90	5	1	2
2	15	2x	No	65	No	Yes	Yes	90	5	4	0
3	10	2x	Yes	15	Yes	Yes	Yes	10	5	4	25
4	15	1x	Yes	65	No	No	Yes	10	1	4	13
5	15	2x	No	65	Yes	Yes	No	10	1	1	6
6	15	2x	Yes	15	Yes	No	Yes	90	1	1	9
7	10	2x	Yes	65	No	No	No	10	5	1	6
8	10	1x	Yes	65	Yes	Yes	No	90	1	4	4
9	10	1x	No	65	Yes	No	Yes	90	5	1	0
10	15	1x	No	15	Yes	No	No	10	5	4	35
11	10	2x	No	15	No	No	No	90	1	4	2
12	10	1x	No	15	No	Yes	Yes	10	1	1	26

Table 3. DoE responses of stray plate

is then obtained.[2] Note that the backside of the stray plate was analyzed, the side that was in direct contact with the soldermask.

Minitab 16® software was used to create the Plackett-Burman 12 run designed experiment, and the design array is shown in Table 2.

When running any designed experiment it's important to do all you can to minimize any noise influence in the testing. Noise, or lurking variables, are simply unidentified variables that change during an experiment that cannot be controlled. Lurking variables can be tolerated when they're managed correctly but disastrous when they're not. To help in reducing noise it's important that the test vehicles mimic the real production pieces, so for this experiment the test vehicles were actual multilayer printed circuit boards, in panel form, with a dimension of 16 x 18 inches. Another critical step in negating noise is to randomize the experimental runs; if noise is present we generally see an inflation of the error variance without adding bias to the real differences between the factor levels.

The responses to each of the 12 individual experimental runs are shown in Table 3. The counts of stray plate are actual individual pieces found on each test vehicle.

Analysis of variance (ANOVA) was used to analyze the full model; Table 4 shows the ANOVA, while Figure 4 shows a Pareto chart of the standardized effects. The final model (reduced) ANOVA and Pareto chart of the standardized effects are shown in Table 5 and Figure 5.

We can see in final model ANOVA

Analysis of Variance for Stray Plate (coded units)						
Source	**DF**	**Seq SS**	**Adj SS**	**Adj MS**	**F**	**P**
Main Effects	10	1421.33	1421.33	142.133	1.67	0.544
Step Exposure	1	0.33	0.33	0.333	0.00	0.960
Developer Passes	1	85.33	85.33	85.333	1.00	0.500
Pre Cure	1	8.33	8.33	8.333	0.10	0.807
Final Cure	1	408.33	408.33	408.333	4.79	0.273
UV Bump	1	75.00	75.00	75.000	0.88	0.521
Pre Scrub	1	0.33	0.33	0.333	0.00	0.960
Cleaner	1	27.00	27.00	27.000	0.32	0.674
Micro Etch	1	736.33	736.33	736.333	8.63	0.209
Pre Dip	1	5.33	5.33	5.333	0.06	0.844
Activator	1	75.00	75.00	75.000	0.88	0.521
Residual Errors	1	85.33	85.33	85.333		
Total	**11**	**1506.67**				

Table 4. ANOVA of the full model.

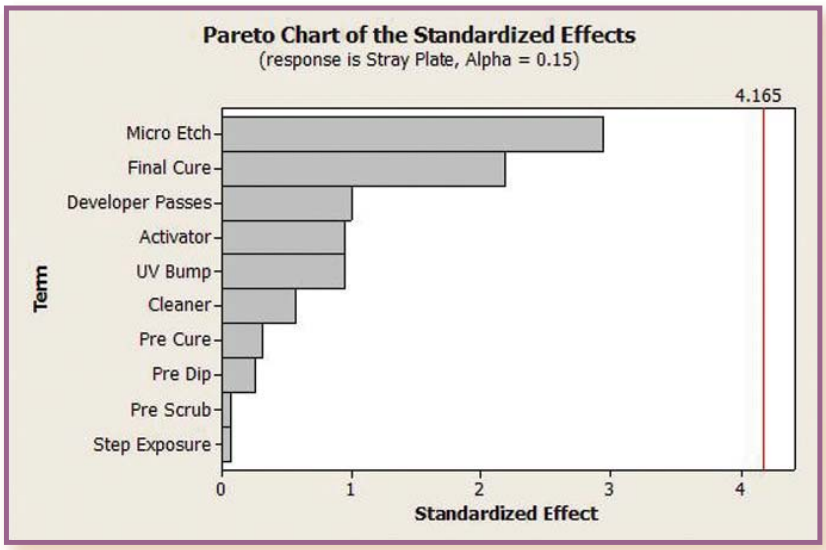

Figure 4: Pareto chart of the standardized effects for the full model.

Analysis of Variance for Stray Plate (coded units)						
Source	**DF**	**Seq SS**	**Adj SS**	**Adj MS**	**F**	**P**
Main Effects	2	1144.7	1144.7	572.33	14.23	0.002
Final Cure	1	408.3	408.3	408.33	10.15	0.011
Micro Etch	1	736.3	736.3	736.33	18.31	0.002
Residual Errors	9	362.0	362.0	40.22		
Lack of Fit	1	225.3	225.3	225.33	13.19	0.007
Pure Error	8	136.7	136.7	17.08		
Total	**11**	**1506.7**				

Table 5. ANOVA of the final model.

(Table 5) that the Lack-of-Fit is statistically significant (p-value significantly below 0.05) which indicates we are lacking higher order terms (that of possible interactions and/or quadratic terms). With Lack-of-Fit detected, the general protocol would be to abandon the tentative regression model in attempts to find a more appropriate equation[3, 4]. Our primary objective is discovering significant factors, and our secondary objective is building a regression model, that of predicting the value of stray plate (dependent variable) from the factors (independent variables), $Y = f(x_1, x_2...x_n)$. Due to the lack-of-fit we will abandon the secondary objective and ignore this condition.[5]

We have detected two statistically significant variables, that of Micro Etch and Final Cure, which deserve further investigation.

Results of the SARIS™ analysis are shown in Figure 6. Of significant interest is the copper (Cu) that was detected. There is high correlation between the copper detected on the backside of the stray plate defect, and the statistically significant factor Micro Etch. Why? Because the Micro Etch is removing copper.

For a reference point the blue soldermask was analyzed by SARIS™, and the results are shown in Figure 7. Note that copper is detected, but at a much lower level than in the stray plate defect, so we can conclude that the low level detected in the soldermask is a filler and the high level detected on the backside of the stray plate is an anomaly.

The screening DoE was able to eliminate a significant number of the study variables, and study variables that are eliminated contain a wealth of information about the process itself and are key in developing enhanced process knowledge. Therefore, it is recommended that time be allotted to debrief team members and those responsible for the processes under study.

Armed with the identification of the two significant factors and the results of the SARIS™ analysis, further investigations were made into the entire upstream processing.

It was found that soldermask operators were slip sheeting and stacking final cured panels as they were coming out of the oven. The slip sheets were investigated as a potential source of contamination. Figures 8 and 9 show what was found embedded in the slip sheets themselves.

Contaminated slip sheets were transferring copper particles to the hot soldermask surface when the panels were stacked after final cure.

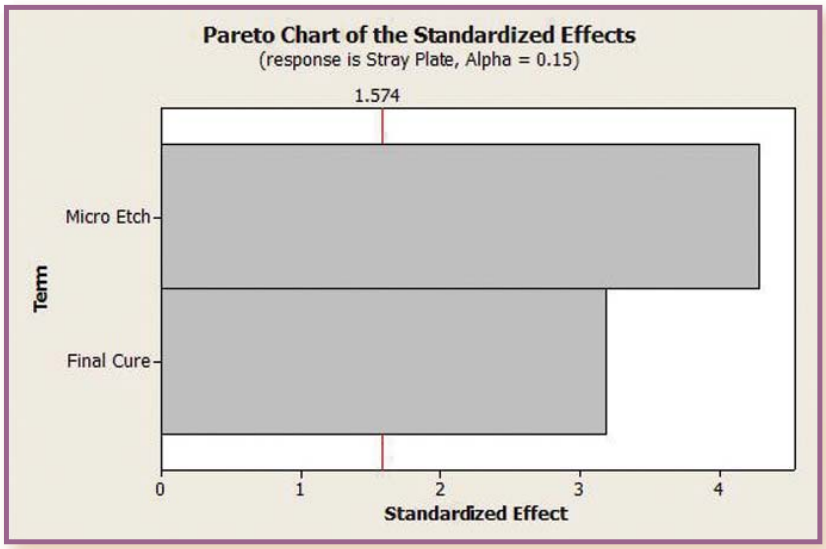

Figure 5. Pareto chart of the standardized effects of the final model.

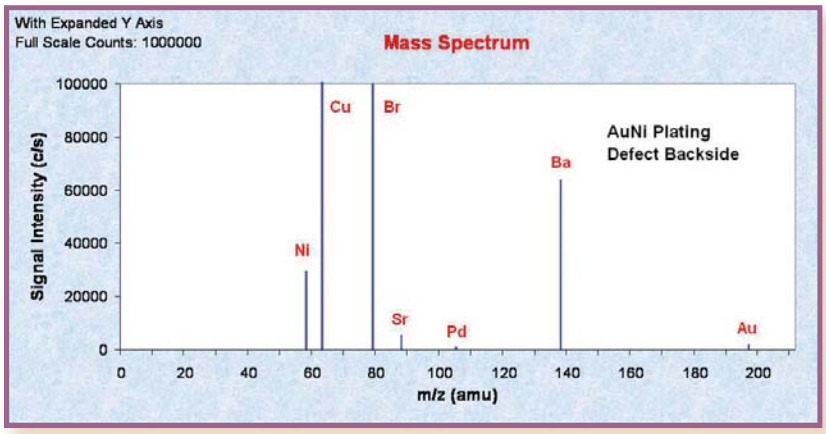

Figure 6. Results of the SARIS™ analysis of the stray plate.

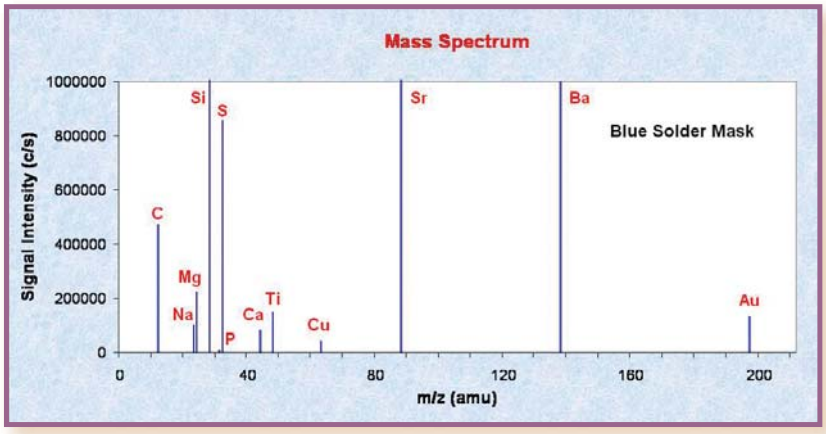

Figure 7. Results of the SARIS™ analysis of the blue soldermask.

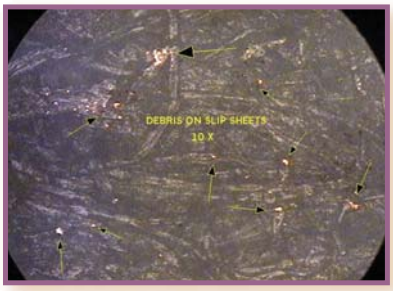

Figure 8. Metallic copper particles embedded in the slip sheets (10x).

Figure 9. Metallic copper particles embedded in the slip sheets (40x).

The more advanced the cross-linking of the soldermask (longer final cure time) the less likely the soldermask was to pick up copper particles, which makes sense. These types of stray defects are usually amorphous in appearance, and once the root cause of the problem was identified and eliminated the stray plating ceased.

Knowledge of the potential for soldermask to pickup particulate should be disseminated throughout the manufacturing organization, and lessons learned should be archived in a FMEA (Failure Mode Effects Analysis). These activities are known as *process management.*

SUMMARY

When trouble arises in today's complex manufacturing processes, the process engineer needs to react quickly. Many times the engineer is faced with dozens if not hundreds of process control options to change. Using designed experiments as a tool for troubleshooting can be very effective, saving both time and money.

The Plackett-Burman family of designed experiments can be an effective tool to aid the process engineer in troubleshooting a process as it allows one to separate the "vital

few" factors from the "trivial many." The process engineer must keep in mind that statistics are only tools to help us; they don't replace the engineers' skill and intelligence.

REFERENCES

1. NIST/SEMATECH (2010). e-Handbook of Statistical Methods.
2. SARIS™ Laser Ablation ICP-MS analytical technique developed by Balazs NanoAnalysis
3. Montgomery, D. (2005). *Design and Analysis of Experiments, 6th Edition*, Wiley & Sons Inc., Hoboken, NJ.
4. Mathews, P. (2005). *Design of Experiments with Minitab*, ASQ Quality Press, Milwaukee, WI.
5. Launsby, R., and Schmidt, S. (2005). *Understanding Industrial Designed Experiments, 4th Edition*, Air Academy Press, Colorado Springs, CO.

BIO

Patrick Valentine is the North American PCB and EP&F Business Manager for OMG Electronic Chemicals. As part of his responsibilities, he teaches six sigma green belt and black belt courses. He holds a Master's Degree in Business from Regent University, Six Sigma Master Black Belt certification from Arizona State University, and is an ASQ certified Six Sigma Black Belt. Patrick can be contacted at patrick.valentine@omgi.com